STRENGTH OF GRANITE SATURATED BY VARIOUS FLUIDS

by

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This thesis describes experiment-based research aiming to evaluate the change of strength of granite saturated by different fluids. The granite specimens are vacuum saturated with water, hydrochloric acid (HCl), sodium hydroxide (NaOH), and kerosene. Then, the flexural tensile strength of granite specimens was measured in three-point bending tests. Results indicate the strength of granite saturated by fluids is decreased compared with dry granite. However, there is no significant difference between these types of fluids. Compared to the dry granite specimens, water, HCl, NaOH, and kerosene reduce the strength of granite by 25%, 32%, 26%, and 24%, respectively. HCl in a range of low loading rate (less than 20 MPa/min) shows a greater strength reduction, but even this effect is relatively small and only marginally statistically significant. Also, the polar fluid (aqueous) does not have a different performance compared to the nonpolar fluid (kerosene), although there is a small difference wherein the kerosene-saturated specimens are very slightly stronger (although of marginal statistical significance) compared with those saturated by aqueous solutions. The experiments suggest that chemical fluid-rock interaction plays a minimal role compared to the mechanical influence of the fluid, for example through lubrication of grain boundaries and/or local pore pressure generation. Initial discussion is provided for this revised hypothesis through demonstration that specimens saturated by more viscous hydraulic oil are consistently weaker than those saturated by less viscous kerosene.

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1.0 INTRODUCTION

1.1 OVERVIEW

Stress corrosion is a material failure phenomenon that combines the influence of stress with a corrosive environment. The first observation of this damage related to environment and stress was shown in the 19th century, and it was called “season cracking” (Walter Rosenhain 1919). Later in the 20th century, more and more materials, such as metals, glasses, ceramics, etc., were shown to be affected by this type of damage, and it was eventually recognized as so-called “stress corrosion”. Stress corrosion commonly leads to small cracks in materials, which are difficult to detect and yet eventually can result in failure.

Granite is one of the most common igneous rock on the Earth. It is widely used in many applications, including bridges, pavements, countertops, and decorations. It is also commonly encountered in subsurface drilling operations. Because of these types of applications, granite is frequently damaged by load and/or the environment. This combined damage leads to granite material failure or cracking with results ranging from the reduction of visual aesthetics to structural collapse. Due to the possibility of severe consequences, it is necessary to have a better understanding of stress corrosion in granite. Accurate understanding of stress corrosion in granite can inform mitigation method to extend its service life and reduce the risk of failure. On the other hand, some situations may require the breaking of granite. One example is drilling of wells and

boreholes. In petroleum engineering, drilling typically encounters sedimentary rocks, however, encounters with granite do occur and these can severely damage drill bits, as shown in Figure 1. Also, in the area of Enhanced Geothermal Systems (EGS), the desire is to drill into and hydraulically-stimulate deep, hot granite formations for the purpose of extracting heat that will be used to generate electricity (see review of Tester 2006). In both cases, granite breakage is essential. Furthermore, various forms of cracking related to stress corrosion and/or thermal cracking have been suggested as forms of reservoir stimulation (e.g. Regenauer-Lieb 2015) and subcritical crack growth, possibly impacted by stress corrosion, has been suggested to be important for prediction of hydraulic fracture initiation granite (Lu 2015).

Figure 1 Damage on drillstring because of granite (from Zhan et al. 2010)

Finally, and perhaps most importantly, both natural and induced seismicity is often associated with rupture of granite. For example, filling of a water reservoir can result in enhanced seismic activities in granitic basement rock which may fail as it is subjected to increased pore water pressure (Liao 1983). More recently, chemical fluid-rock interaction has been proposed as a potential mechanism increasing the risk of seismicity associated with a subsurface injection of CO_2, and/or with wastewater from petroleum extraction (Kim 2013, White 2016).

1.2 LITERATURE REVIEW

1.2.1 Granite properties

Research on granite dates back at least to the nineteenth century. Granite is a common igneous rock. Based on the 2485 chemical analyses, granite, on average, contains 71.83% SiO_2, 14.43% Al_2O_3, 4.10% K_2O, 3.71% Na_2O, and some other oxides (Best 2003). Stress corrosion experiments have also been done on various rocks. Atkinson (1979) stated that the water could decrease the strength of quartz by using the double torsion. Later, Atkinson (1981) showed that pH of the aqueous solutions also has an influence on strength of quartz. Stress corrosion influence on other types of rock is also reported (see Atkinson 1981). However, due to the formation and composition of granite, it is commonly thought that granite is relatively chemically inert and has low porosity, and thus may not be as fluid sensitive as other rocks. Thus, important differences between stress corrosion influence on granite and other types of rock may exist.

1.2.2 Stress corrosion

Stress corrosion refers to the phenomenon whereby materials are degraded by the combined effects of a corrosive environment and tensile stress (Sieradzki 1987). It is a common and sometimes destructive process in engineering materials. Hoar (1963) considered four types of penetration that can be generated when stress is applied to a material. Stress corrosion mainly builds a crack, which is the right-most penetration shown in Figure 2. It is thinner and longer than other types of penetration. Thus, it is easier to reach the boundary of the other and lead to the failure of materials.

Research on stress corrosion firstly focused on metal materials, since the earliest observation of stress corrosion was associated with metals such as aluminum, magnesium, and titanium (Anderson). The mechanism of stress corrosion is not unified, but there are some general theories that have been proposed:

(1) The susceptible path theory proposes that a path within the material is built by stress that could lower the material strength (Mears 1944). This path has an electrochemical potential difference around the material in certain environments, which then enhances corrosion and causes cracking (Fontana 1954).

(2) The hydrogen embrittlement theory suggests a cycle is built in stress corrosion whereby hydrogen ions are taken into the tip of the crack, enhancing crack extension, after which even more hydrogen ions are taken into the extended crack (Edeleanu 1951).

(3) The film theory, which also relates to electrochemistry, states that a protective film that initially coats material surface is broken by stress. This small break leads to a potential difference compared to the unbroken part, which then promotes the crack extension.

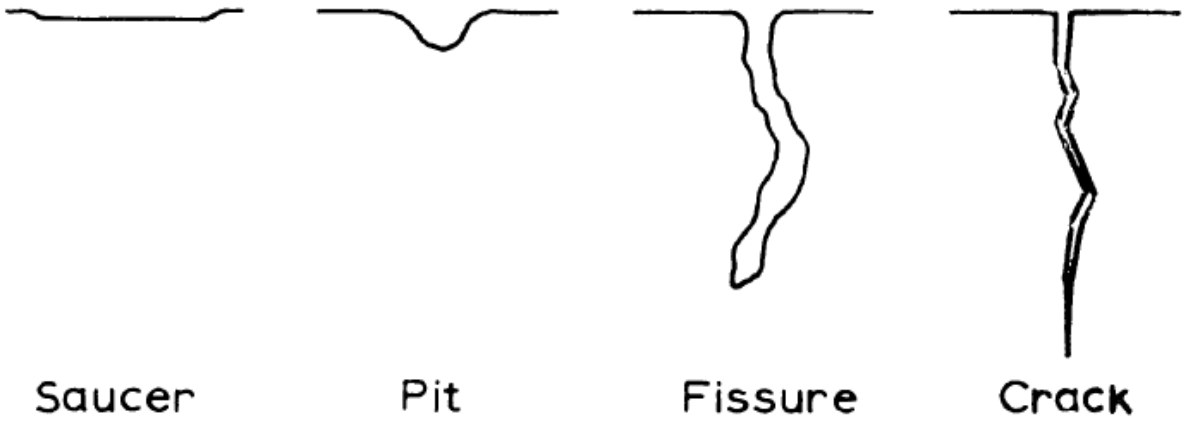


Figure 2 Types of penetration (from Hoar 1963)

Following the work on metals, it has been discovered that silica glass is also sensitive to stress corrosion. It is found that water has an influence on the strength of silica (Charles 1958), and a relationship among crack velocity, applied force, and humidity is found and developed, as shown in Figure 3 (Wiederhorn 1967; Freiman 1985). It shows that the higher humidity leads to a faster crack velocity under the same applied force; in other words, water can decrease the strength of glass, and the specific water concentration influences the result.

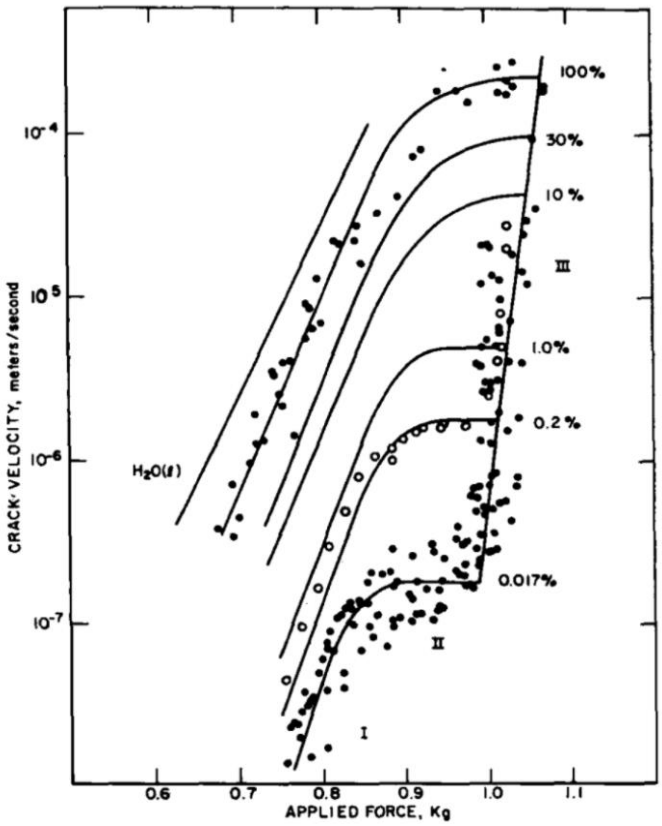


Figure 3 Dependence of crack velocity in glass on applied force (from Wiederhorn 1967)

It is also worthy to note that pH has an influence on strength of glass. In contrast to the conventional thought that glass is chemically inert, low pH aqueous solutions, like hydrochloric acid, decrease the strength of glass, while high pH aqueous solution increase strength of glass, as shown in Figure 4 (Wiederhorn 1973). It also observed in Figure 4 that the crack velocity in specimens attains to an asymptotic value when stress intensity factor is increasing. Motivated by such observations, extensive research has been done to explore the mechanisms and origins of water influence on

silica glass. Many of these studies rely on a particular hydrolysis reaction (Figure 5, left) (Mould 1961; Ito 1981; Michalske 1982), namely

$$\equiv \mathrm{Si} - \mathrm{O} - \mathrm{Si} \equiv + \mathrm{H2O} \leftrightarrow 2 \equiv \mathrm{SiOH} \quad \textbf{(1-1)}$$

Kohn (1989), then, suggested a theory that hydrogen atoms in the H_2O group are attracted by oxygen atoms in SiO_2, and oxygen in H_2O is attracted by silicon because of dipolar interaction (Figure 5, right). The result is an increased ability to break Si-O bonds in the presence of water.

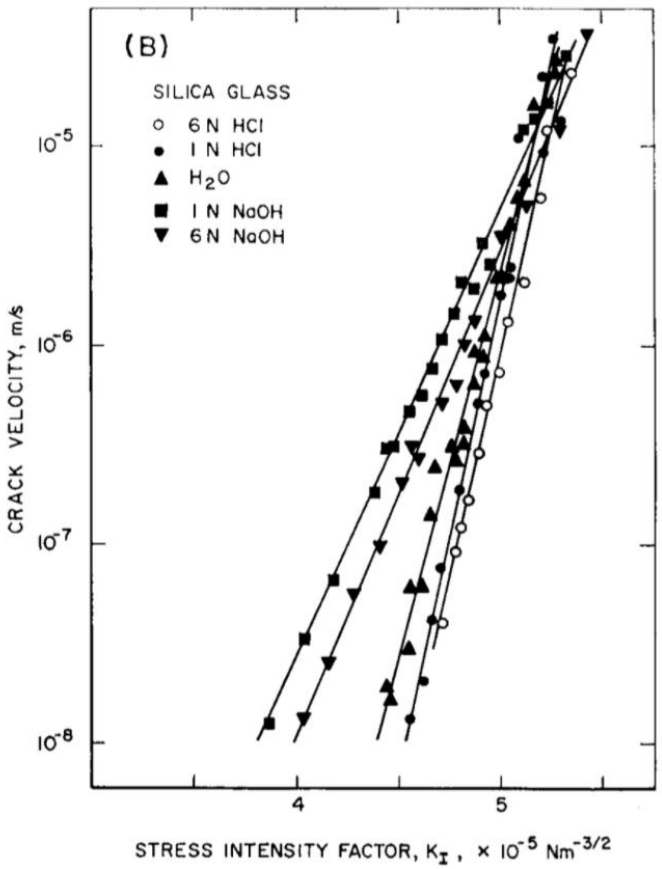


Figure 4 Crack velocity test in glass for different aqueous solutions (from Wiederhorn and Johnson 1973)

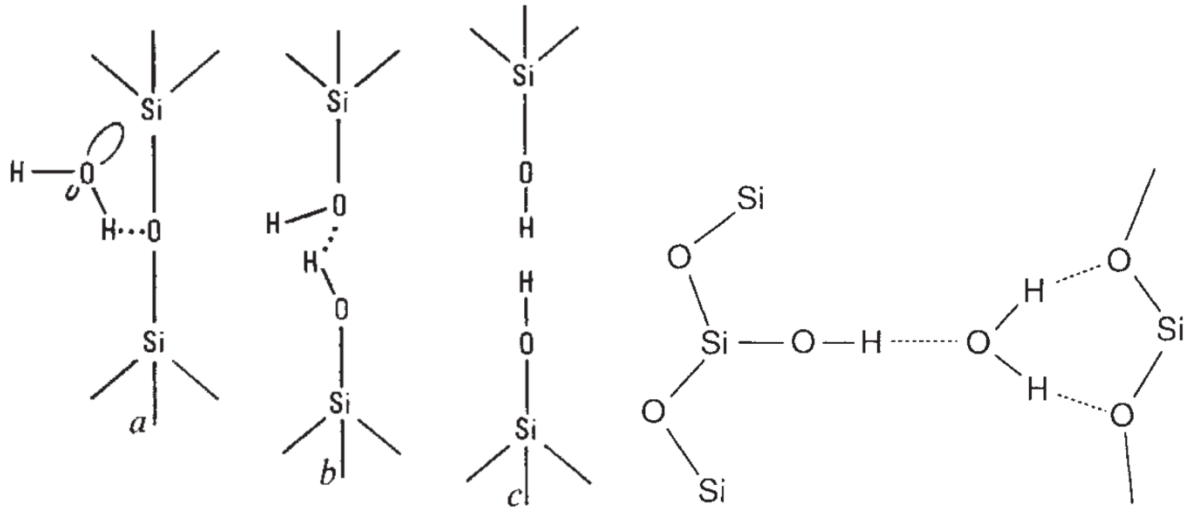


Figure 5 Dipolar interaction of water and silica (after Michalseke and Freiman 1982 and Kohn et al. 1989)

Rock is a more complicated system compared to silicate glass or metals. The research on stress corrosion of rock was less active compared to metals, glasses, and ceramics during the early days of stress corrosion studies. But it was soon realized the importance of studying the cracking and strength characteristics of rock for improvement of rock stability in petroleum, civil, and mining engineering. The early works focus on rock with simple composition, such as quartz. Atkinson (1981) observed that the NaOH solution weakens quartz, and HCl solution, on the other hand, strengthens quartz. Later, El-Shall (1984) showed that a surface-active chemical environment promotes crack growth in quartz, that is, decreasing the strength.

Later, research on strength of complex, quartz-rich sandstones gave some different observations in some ways at odds with earlier work on quartz. The research by Mann (1960) suggested that the strength of sandstone depends on the abundance of clay minerals. Specifically, they observed that after being saturated with water, the strength of sandstone is reduced by 8% to 20%. Also, they suggested that the strength reduction of sandstone is in proportion to the abundance of clay

minerals. This research shows the important influence of clay minerals on strength of sandstone, and this result may lead to theories of strength reduction related to intergranular lubrication (owing to the dependence of clay consistency on water content). Later experiments by Feucht (1990) showed that water reduces the frictional strength by 33% compared to dry specimens. In these experiments, pH and ionic concentration are also controlled. The results (Figure 6) show that NaCl solution with low ionic strength (0.2 M) produce less reduction of material strength than high ionic strength (5 M). It also needs to be noted that pH performs an important role, decreasing strength in both low and high pH environments, most significantly in high pH environments.

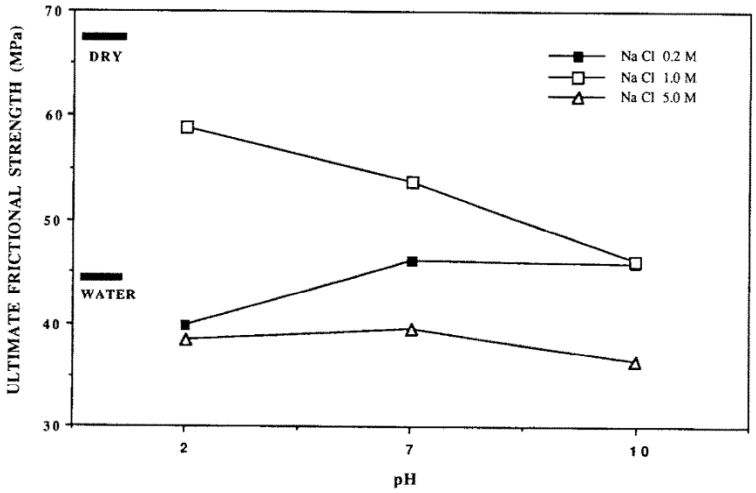


Figure 6 Relationship between frictional strength, pH, and ionic strength for sandstone (from Feucht and Logan 1990)

1.3 MOTIVATION

Although many experiments and studies have been conducted on stress corrosion of metals, silica glasses, and several kinds of rocks, a knowledge gap exists for an understanding of stress corrosion in complicated rock materials, especially granite.

One may be tempted to assume if a type of rock with very low porosity and relative chemical inertness will have little influence from the fluid since the previous research did not test the strength of rock with very low porosity. The rock used in the previous research includes calcite rock with a porosity of 5.3% (Rutter 1972), sandstone with a porosity of 21% (Menéndez 1996), and limestone with a porosity of 3% (Baud 2000). However, when considering a granite specimen, it has a porosity between 0.4% to 1.5%, so that granite will absorb much less fluid than most other types of rocks. Therefore, if there is some reaction threshold, the rock saturated with fluid at a concentration lower than the threshold may not reduce in strength as much as other rocks. The research on granite saturated with various fluids may give a better understanding of stress corrosion of low porosity rocks. The benefit from the result may also enable the risk assessment of granite used in infrastructure and applications in hydraulic fracturing, especially for geothermal reservoirs.

The previous research paid attention to stress corrosion associated with carbonate and silicate rich rocks. However, most of these rocks have either acidic or alkaline properties. For instance, limestone mainly contains calcium carbonate ($CaCO_3$), which makes it easier to be influenced by acid. Furthermore, sandstone contains on average 79% silica (SiO_2) and less than 5% alumina (Al_2O_3), so its strength could therefore be proposed to decrease in relation to strength of the acid

(Leith 1951; Feng 2004). On the contrary, granite has more complex mineralogy and microstructure compared to many other rocks. The main chemical composition of granite is more than 70% silica and 15% alumina, which brings potential for sensitivity to both acidic and alkaline conditions. Meanwhile, because of the low porosity of granite, specimens are saturated by relatively less fluid compared to other rock types. With these differences in mind, it is an unknown if granite rock has similar sensitivity to pH changing as other rocks.

It is also worth noting that most previous experiments are based on water or aqueous solutions. This is true for experiments that are designed to use a salt solution such as NaCl, $CaCl_2$, and $NaHCO_3$ (Feucht 1990), alkalis and acids such as HCl and NaOH (Atkinson 1981), and river water (Feng 2004). All consider water-based fluid. Water is one of the most polar of the common fluids. The previous experiments have already shown that water or water-based fluids have an influence on the strength of silica and silica rocks, and proposed that the effect is, at least in part, because of its polarity (Ito 1981). However, there are few experiments considering a strength reduction due to non-polar fluid, and it is still unclear if the non-polar fluid has a similar impact on strength reduction. If nonpolar fluid leads to similar strength reduction, it may suggest that the dominant factor of strength reduction is not primarily due to the promotion of breakage of silica bonds.

1.4 RESEARCH OBJECTIVE

The thesis research project investigates the nominal tensile strength of granite saturated by different fluids: water, hydrochloric acid, sodium hydroxide, and kerosene. Thus, the goal of the research is to find the change of strength when granite is saturated by those fluids, and the specific research objectives are as follows:

- Test the hypothesis that the strength of granite specimens saturated by fluid will be lower than the dry granite specimens.
- Test if the pH of the fluid has a substantial influence on the strength of granite.
- Compare the strength of granite saturated by non-polar and polar fluids, where all fluids have similar viscosity and density.

2.0 EXPERIMENTAL PROCEDURE

This chapter introduces the details of the experiments as well as the related specimen preparation processes, specifically: (1) specimen selection, (2) preparing the fluids, (3) preparing the specimens by saturating with the desired fluid, (4) description of apparatus, and (5) experimental procedure.

2.1 SPECIMEN SELECTION

The rock chosen for the experiment is called Pegasus Beige Granite. It is a common granite, widely-used in industry including for granite countertops. The granite used in the experiments was commercially produced, supplied as 518 mm × 102 mm × 19 mm slabs, and polished on the top face. Because uniformity is essential to its commercial use, it is reasonable to consider the specimens to have reasonably equivalent properties.

2.2 FLUID PREPARATION

The fluid used in the experiment will be absorbed into the pore space of the granite. There are four types of fluid that are used: (1) water, (2) 10% by weight hydrochloric acid (HCl) (3) 10% by

weight sodium hydroxide (NaOH), and (4) kerosene. The hydrochloric acid originally is 38% weight percent and is subsequently diluted to 10% by weight. The high concentration hydrochloric acid is volatile, so the entire process is performed in a fume hood. In addition, sodium hydroxide supplied as 50% by weight is diluted to 10% by weight. Table 1 shows the comparison of density and viscosity. The intent is to isolate the role of fluid chemistry by maintaining the density and viscosity of fluids in the range of 0.8 – 1.0 g/cm^3 and 0.89 – 1.86 cP, respectively.

Table 1 Density and viscosity of fluids used in the experiments

Chemicals	Density (g/cm^3)	Viscosity (cP)
Water	1.00	0.89
10%wt HCl	1.05	1.16
10%wt NaOH	1.11	1.86
Kerosene	0.81	1.64

2.3 SAMPLE PREPARATION

2.3.1 Beam preparation

To facilitate three-point bending tests, the granite slabs are saw-cut into 0.75-inch × 0.75-inch × 3.5-inch (19.1 mm × 19.1 mm × 88.9 mm) beams as shown in Figure 7.

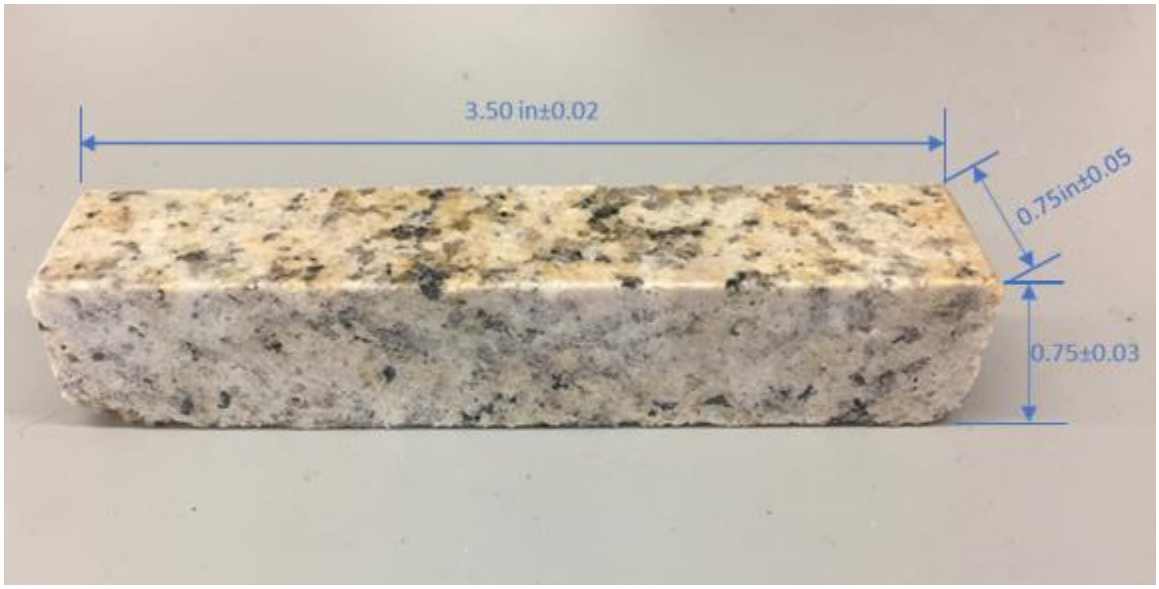


Figure 7 Specimen dimensions

2.3.2 Fluid saturation

After cutting, the granite beams need to be saturated with the desired fluids. Saturation follows a vacuum method, as described by Lisabeth (2015). As shown in Figure 8, the granite is first

immersed in the fluid. Then the entire specimen container is placed in a vacuum chamber and is kept under vacuum for 24 hours. The vacuum is then released, and the specimen is left in the fluid for an additional 24 hours. Note that upon applying the vacuum to the chamber, some bubbles appear on the surface of the fluid. Normally, the bubbles appear smoothly, however, for the sodium hydroxide, the bubbling is much more violent than for the other three fluids, like boiling water. It is reasonable to infer that the sodium hydroxide may have a reaction with the granite that produces a volatile product.

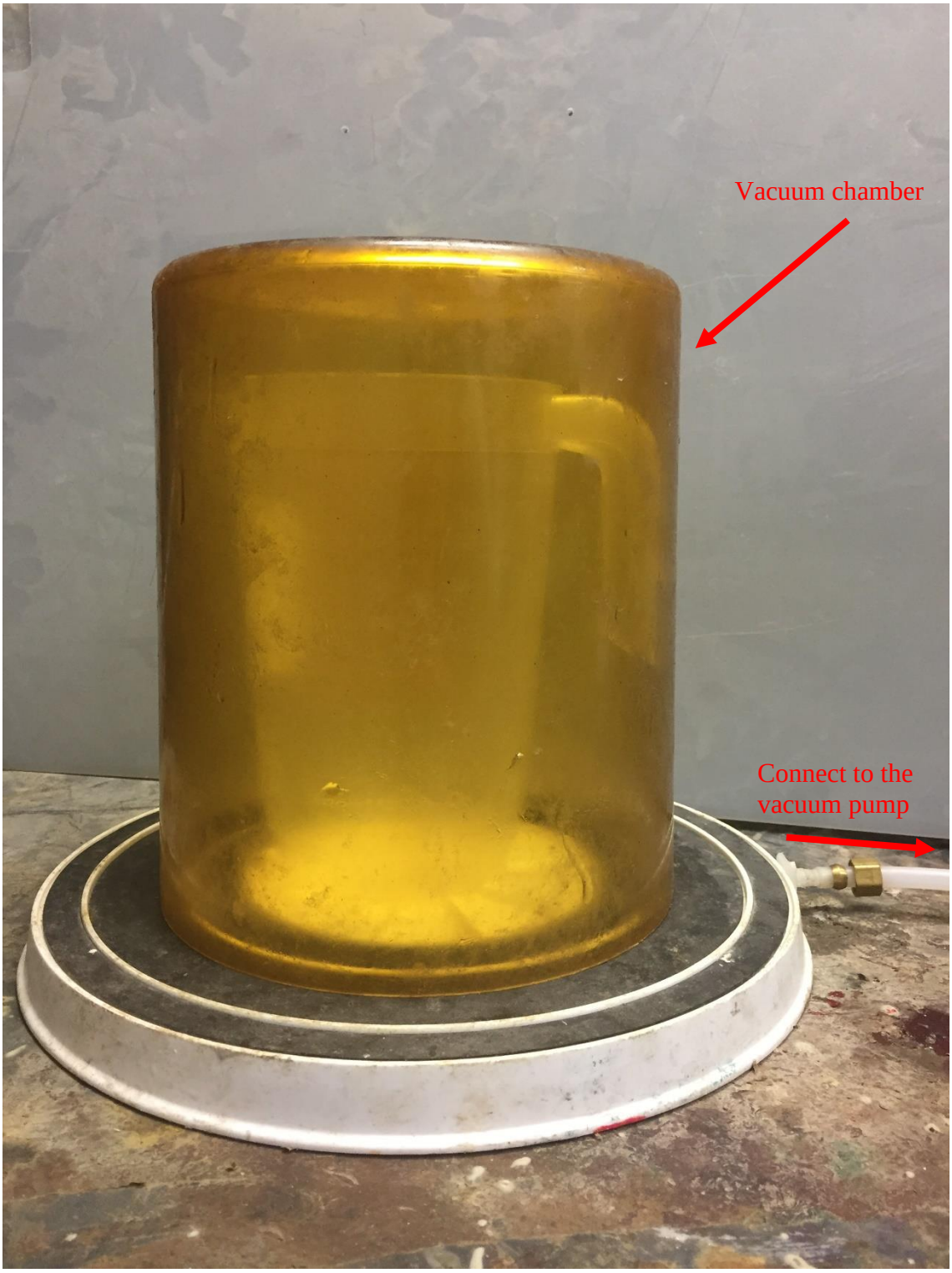


Figure 8 Vacuum chamber setup

The mechanism of the saturation process is to firstly evacuate the air from the pore space. Then, after stopping the vacuum, the atmospheric pressure will push the fluid into the pores because of the pressure difference. The saturation process has two variants, which are here called the short-time and long-time process. For the short-time process, the vacuum environment is maintained until there are no bubbles that appear, and then allowing the granite beams 12 hours to absorb the fluid under atmospheric pressure. For the long-time process, the vacuum application is kept the same, but absorption under atmospheric pressure is extended to 24 hours. Because of the difference between two absorption times, the saturation of the two groups of granite will be distinct, so it is tested if the saturation time has an influence on tensile strength in the experiment (See Results section). After the saturation process, the hydrochloric acid is neutralized before disposal.

2.4 APPARATUS

Loading is provided by a small-size load-frame (Figure 9). The load is generated by pumping hydraulic oil into the actuator in the range from 0.07 MPa to 21 MPa. The pump used to apply the pressure to the actuator is a 260D ISCO syringe pump. The recorded data includes time, fluid pressure, and the output of the load cell used to determine the force applied to the specimen. Note that 0.07 MPa to 21 MPa of fluid pressure generates 13 to 4092 Newtons of force from the hydraulic actuator used in these experiments.

The specimens are tested in a classical three-point bending configuration (for further description, see e.g. Mazel 2014). The sample was put on the two support pins, where are 88.9 mm (3.5 inches) apart, as shown in Figure 10. The loading rod above the specimen is held in a groove on the surface of the loading platen, so that the loading point will accurately remain in the same place, thus reducing potential for experimental error.

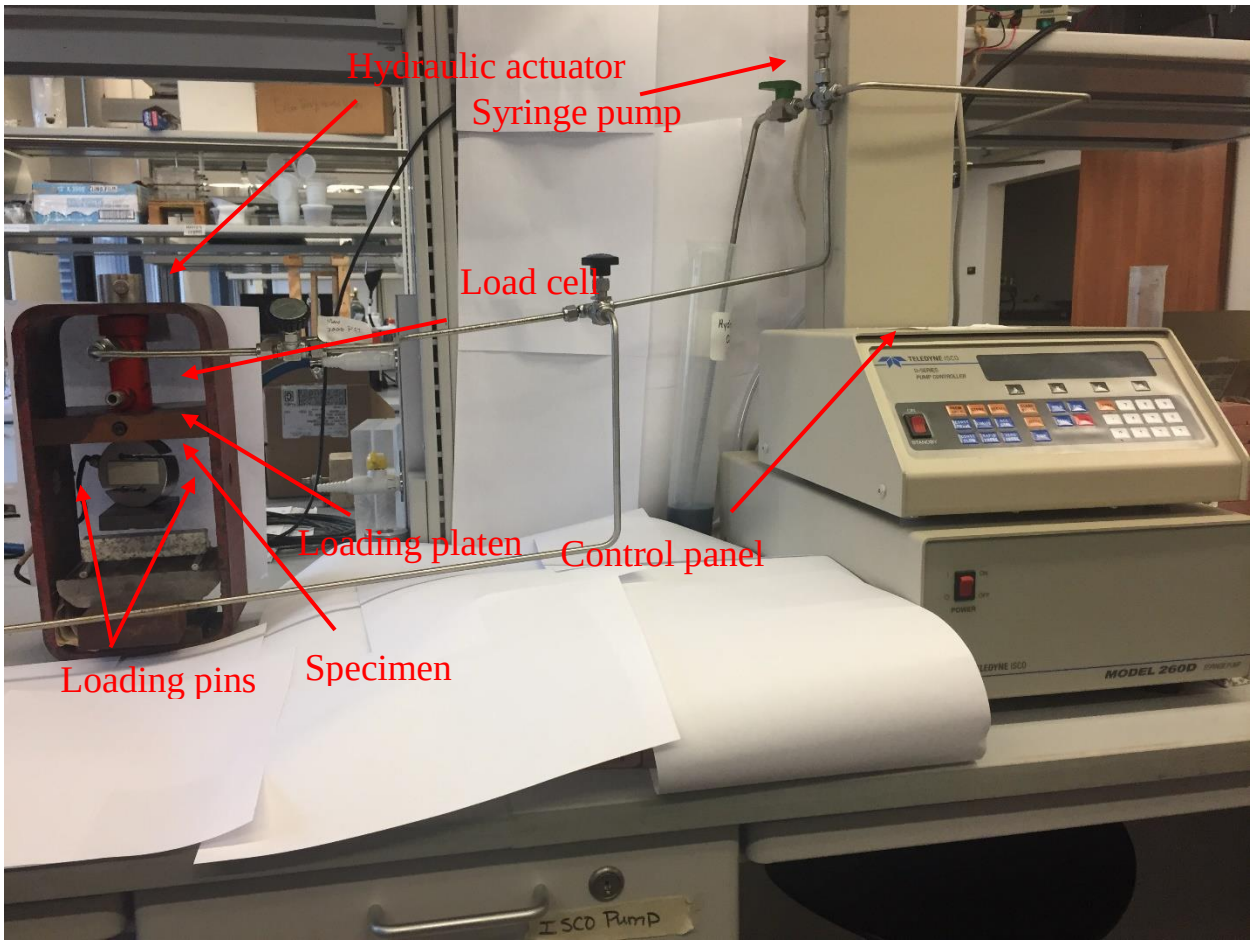


Figure 9 Photograph of experimental apparatus

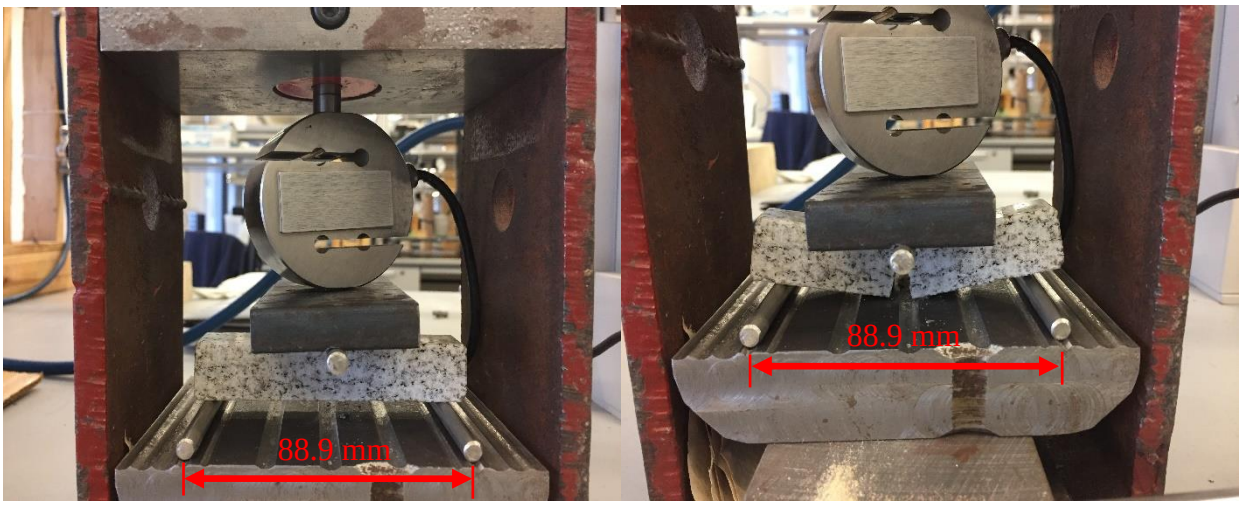
Loading the specimen in this way generates compression on the top of the specimen and tension on the bottom of the specimen. The maximum tension is generated on the bottom surface, at the midway point between the support pins. This stress is computed as (ASTM):

$$\sigma = \frac{3PL}{2dh^2} \quad \textbf{(2-1)}$$

where σ is tensile stress, P is applied load, L is specimen length, d is specimen width, and h is specimen height.

2.5 EXPERIMENTAL PROCEDURE

The samples are placed on support pins with a span of 88.9 mm (3.5 inches), as shown in Figure 10a. An initial load of 222 N is then applied. This load has two purposes. Firstly, it fixes the sample in place so that the increasing load will not move the sample when applied. Secondly, this seating load gives time to move to a safe distance before beginning to apply the high pressure. The increasing load is then applied to the specimen with a constant rate of increase (linear ramp). The pumping process, as well as the data acquisition, ceases when the specimen breaks, as shown in Figure 10b. Fernau (2016) demonstrate that the apparent strength of granite in three-point bending depends on the loading rate. To account for this dependence, loading is set at several rates between 0.34 MPa/min and 33 MPa/min. These rates lead to total test times between 4 and 360 seconds.



(a) (b)

Figure 10 Prepared sample a) before and b) after the experiment

The experimental program consists of carrying out the procedure for: 1) dry specimens. 2) water saturated specimens, 3) HCl saturated specimens, 4) NaOH saturated specimens, and 5) kerosene saturated specimens.

Note that the dry specimens comprise a repeat of the tests reported by Fernau (2016). Thus, these allow a check of consistency of material and procedure.

2.6 STATISTICAL METHOD

The data obtained from the experiments are compared among types of fluid by using *t* test as implemented in the IBM SPSS Statistics 25. The most important value in this table is the significance level (SIG), which also called the P-value. This number determines the confidence level by which two data sets are deemed significantly different. The significance level is in a range

of 0 to 1, corresponding to an estimate of the probability that the data was actually drawn from a single underlying population. Normally, if the significance level is less than 0.05 or 0.1, depending on confidence requirement, it is deemed that two data sets are significantly different.

3.0 EXPERIMENTAL RESULTS

3.1 DRY SPECIMEN GROUP

There are 20 experiments conducted to determine the consistency of current granite specimens and the granite specimens used by Fernau (2016). This experimental group repeated the same process and compares with the results from the previous experiments. The results with loading rate in a range of 1 MPa/min to 150 MPa/ min (145 psi/min to 21756 psi/min) are plotted in Figure 11. Based on nominal stress at failure, the data points can be separated into three groups, and most of the data points of the dry specimen group are in the range of 6 MPa to 11 MPa nominal stress at failure. From 1 to 10 MPa/min, 4 data points drop in the range of 6 MPa to 8 MPa; whereas, from 10 MPa/min to 100 MPa/min, 9 points lie in the range of 8 MPa to 11 MPa. It is also noted that two points have nominal stress at failure greater than 11 MPa. Although the points are scattered, the strength of granite specimens shows a low sensitivity to the loading rate in the range of 1 to 40 MPa/min. Above 40 MPa/min, the nominal strength becomes more dependent on loading rate compared to lower loading rates.

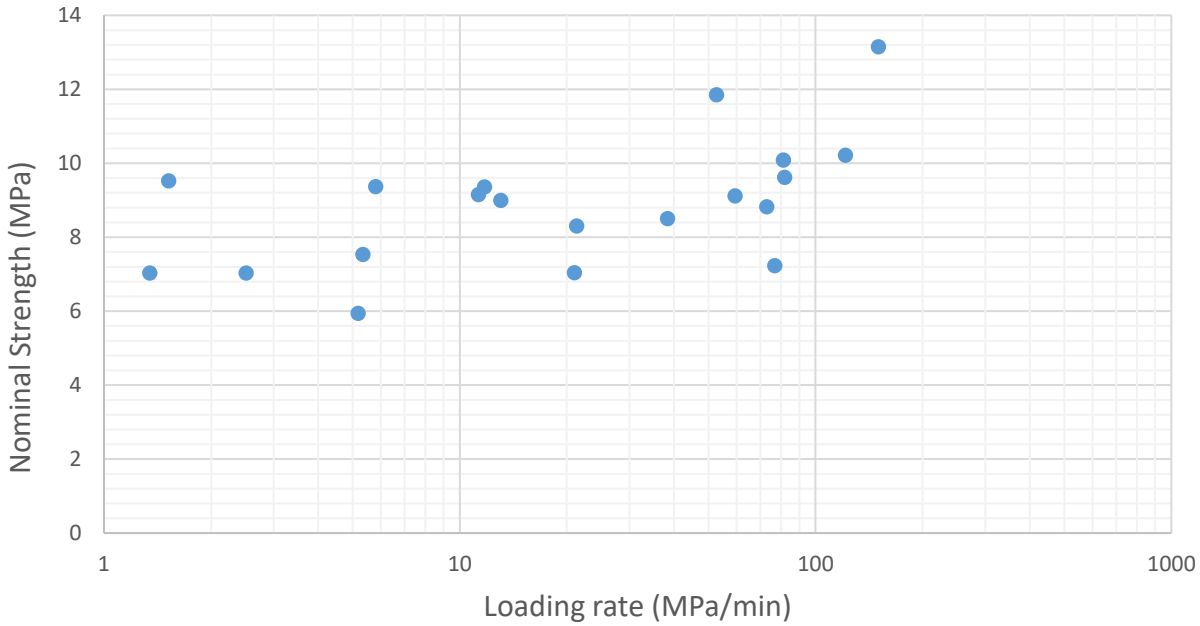


Figure 11 Dry experimental data

3.2 WATER SATURATED GROUP

Observations of 26 granite specimens saturated with water and made to fail under various loading rates shows the influence of water on the strength of granite. The range of loading rate is approximately from 1.5 to 170 MPa/min, corresponding to the pump pressure loading rate range from 1 MPa/min to 134 MPa/min (145 psi/min to 19435 psi/min). It is noted that the data points are scattered, but still it is clear to observe that the data are separated into two parts, as shown in Figure 12. The first part is from a loading rate of 1 to 33 MPa/min, and the second part is from 26 MPa/min to 134 MPa/min. The first part does not show substantial dependence between nominal strength and loading rate, and most points are concentrated in the area of 5 MPa to 8 MPa. The

difference in the second part is that the nominal strength shows a strong dependence on loading rate. From 26 MPa/min to 134 MPa/min, the nominal strength is increased from 3.6 MPa to 10.8 MPa.

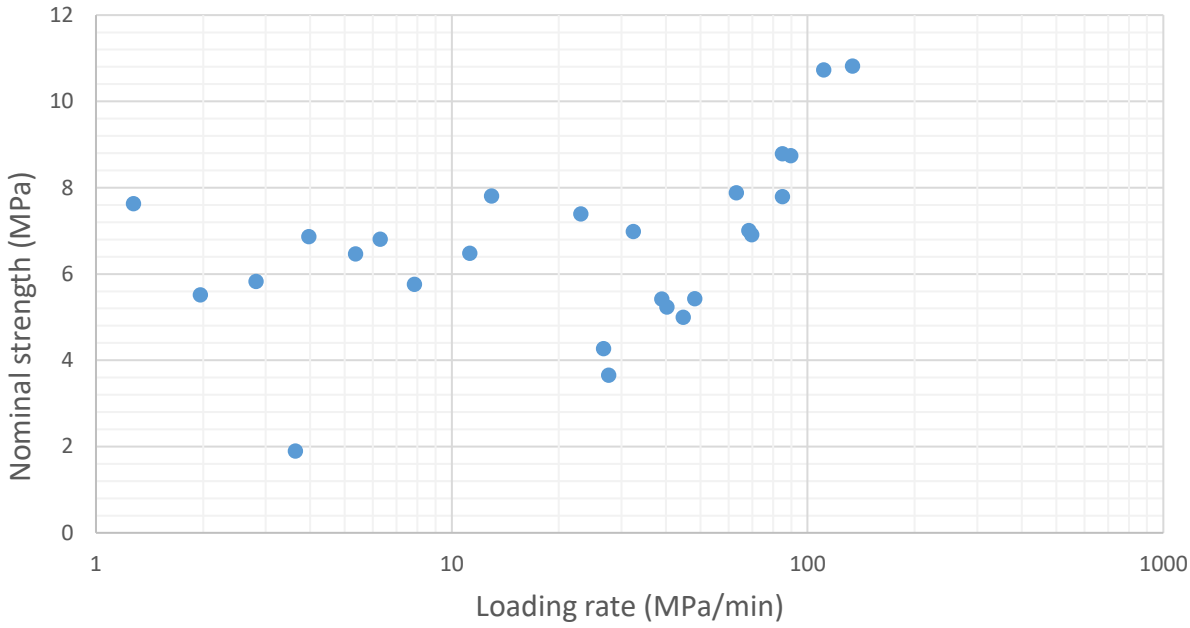


Figure 12 Water experimental data

3.3 HCL SATURATED GROUP

A total of 30 experiments are conducted studying the HCl influence on strength of the granite specimens. Of these, 14 are vacuumed for 12 hours and saturated for another 12 hours ("short time"), and 16 of them are saturated by 24 hours in vacuum followed by 24 hours at atmospheric pressure ("long time"). The 10 percent by weight hydrochloric acid is used in this experimental

group. Triangle markers in Figure 13 show the results of the HCl long-time process experimental group has two parts. It is obvious that the first part, at lower loading rates, is still not very sensitive to the loading rate, but possessing a lower average nominal strength range compared to higher loading rates. From 1 to 17 MPa/min, there are 7 data points in the range of 3.3 MPa to 5.4 MPa. In contrast, above 17 MPa/min, the strength achieves a maximum of 9 MPa and minimum of 4.5 MPa.

Additionally, the sensitivity to saturation time is also tested by changing the vacuuming and saturating time. Provided by Figure 13, the difference is minor, showing most of the nominal strength data points at lower loading rates are very slightly greater for the 12 hours group compared to the 24 hours experimental group. Nonetheless, the difference between the two groups, and hence the dependence of the results on saturation time, is marginal and arguably negligible.

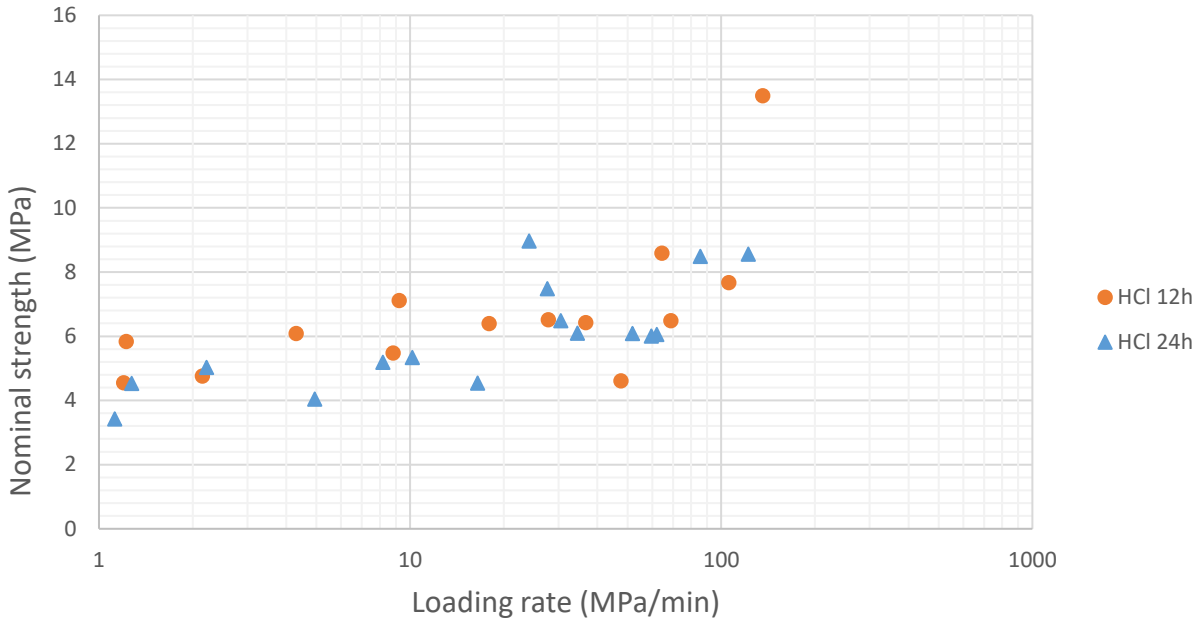


Figure 13 HCl experimental data

3.4 NAOH SATURATED GROUP

At the high end of the pH scale range, the influence of pH on the strength of granite is examined via finishing 29 tests. Similar to the HCl cases, there are 14 experiments performed in specimens subjected to vacuum and saturated in NaOH solution for 12 hours, and 15 experiments performed in specimens subjected to vacuum and saturated in NaOH solution for 24 hours. Dots and triangle markers in Figure 14 show the results of the experiments. Most of the data points from 1 MPa/min to 10 MPa/min loading rates fall in the range from 4 MPa to 6 MPa and begin to depend on loading rate beyond 11 MPa/min. Results do not show obvious differences between two groups of experimental data, i.e. there is no observed dependence upon saturation time.

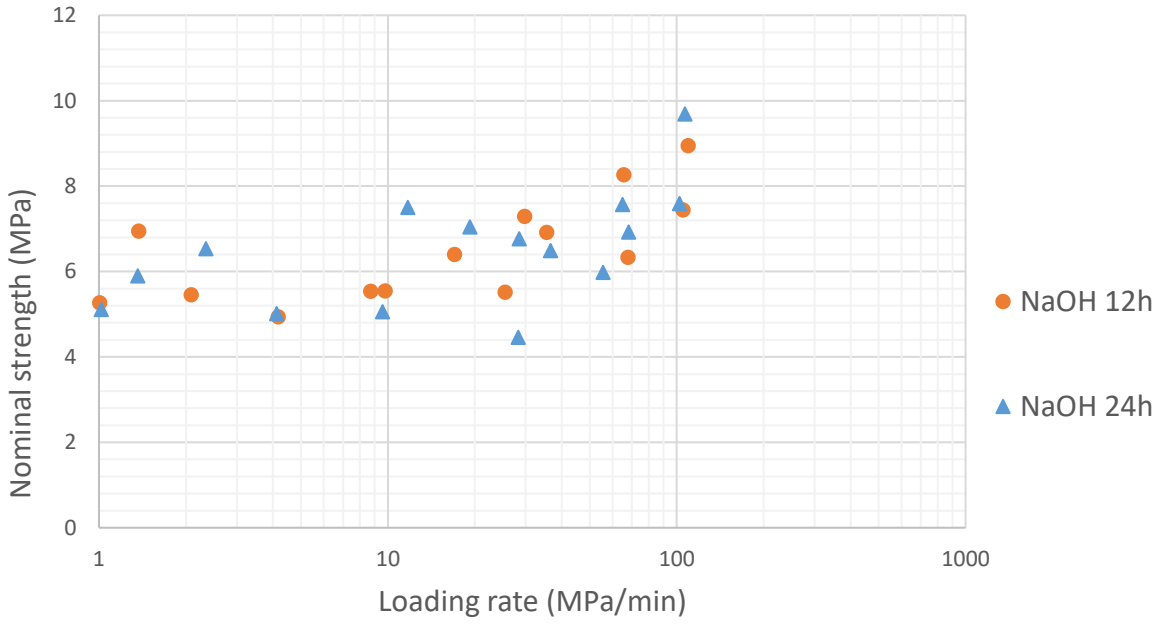


Figure 14 NaOH experimental data

3.5 KEROSENE SATURATED GROUP

The final experimental group tests influence of kerosene on granite specimens. There are 19 experiments using granite specimens saturated with kerosene, and the results are shown in Figure 15. A rate-independent region is observed similar to the water groups. It starts with a loading rate of 1.3 MPa/min to 28.4 MPa/min with nominal strength in the range of 5 MPa to 7.6 MPa. For loading rates above 41 MPa/min, nominal strength shows a strong dependence on loading rate and it increase from 4.6 MPa to 10 MPa.

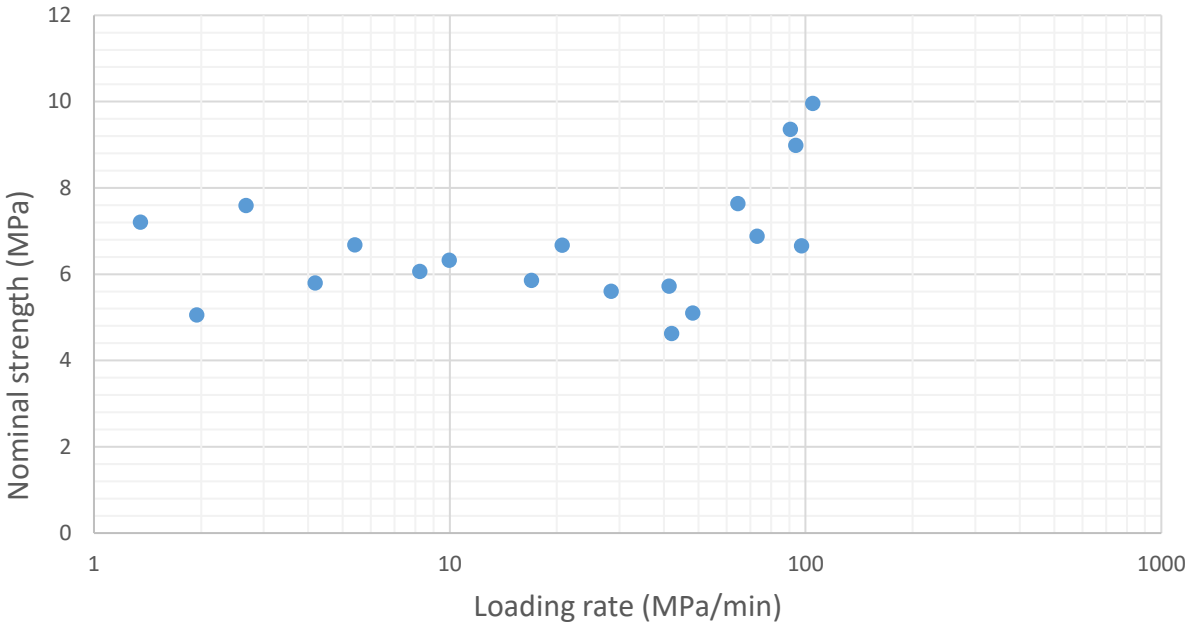


Figure 15 Kerosene experimental dat

4.0 COMPARISONS OF STRENGTH AMONG GROUPS

In this chapter, the data from the different experimental groups will be compared to each other. Meanwhile, the *t* test will be applied to every two datasets by using the IBM SPSS statistics 25 to determine if differences among groups are statistically significant. The null hypothesis for the *t* test is as follows: The two data sets are drawn from the same underlying distribution (i.e. mean and standard deviation is the same). Reported significance levels, then, indicate probabilities that the null hypothesis is true meaning that saturating fluid does not greatly influence the strength of granite specimens.

4.1 DRY EXPERIMENT GROUP

Data from Fernau (2016) was plotted with the data of dry experimental group in Figure 16. It shows that the R square value for linear regression for both sets of dry granite data is very low, owing to the weak correlation of strength with loading rate compared to the scatter of the data. However, it still can be observed that the two datasets have a similar regression equation and they have the similar data scattering range. It is thus possible to say that these two groups of tests are consistent with one another.

To quantify this observation, the statistical method *t* test was applied to the two datasets to compare their difference. From the SPSS result, significance level is equal to 0.23, thereby

statistically justifying the acceptance null hypothesis that the two datasets have no significant difference. This result is not highly confident due to the complex structure of granite but considering the needing of the entire experiments, it is enough to show the similarity. Hence, consistency of materials and methods with Fernau (2016) is confirmed.

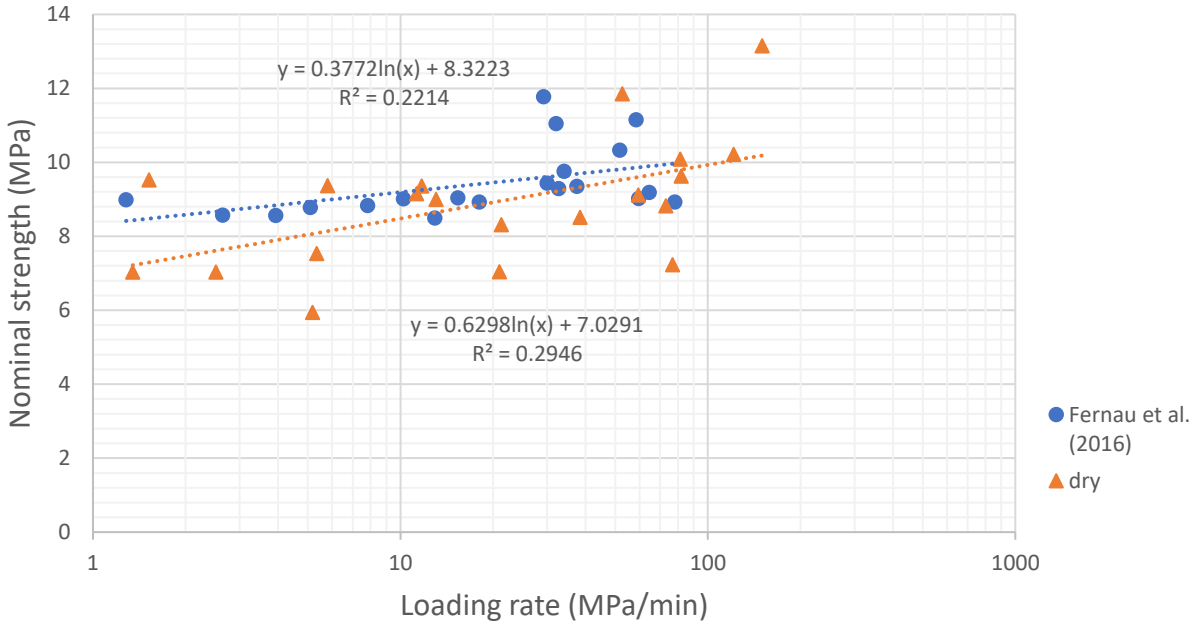


Figure 16 Dry group compared with previously published results (prior data from Fernau et al. 2016)

4.2 DRY VERSUS WATER COMPARISON

Comparing the water experiment results with the dry experiments in Figure 17, it is observed that the best-fit lines for water and dry groups have the similar slopes but the different intercepts. Based on the fitted equation, the failure stress at 1 MPa/min loading rate of granite saturated by water decrease from 7.03 to 4.77 MPa. The change thus shows that the strength of granites after saturation by water compared to dry granite is reduced by around 30%.

Similarly, the *t* test method was applied to the data. The significance level of this experimental group compared to the dry group is smaller than 0.001, which means a very high confidence to reject the null hypothesis. Granite specimens saturated by water have lower strength compared to the dry specimens with strong statistical significance.

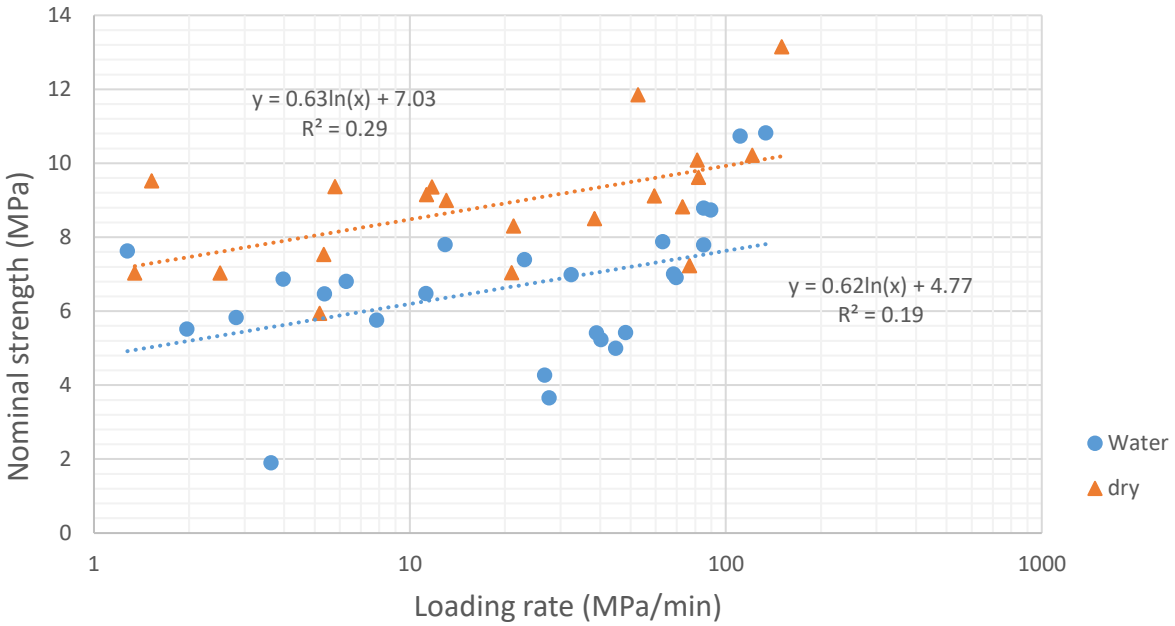


Figure 17 Water group compared with dry group

4.3 PH COMPARISON

A comparison among water, HCl, and NaOH groups are shown in Figure 18. It is observed that the line for NaOH is approximately parallel to the water group, but the HCl group differs. For smaller loading rates, lower than 2.15 and 2.75 MPa/min for water and NaOH, respectively, the failure stress of granite saturated by HCl is the smallest of the three groups. In contrast, somewhat higher stress is required to break the hydrochloric acid saturated granites under higher loading rates.

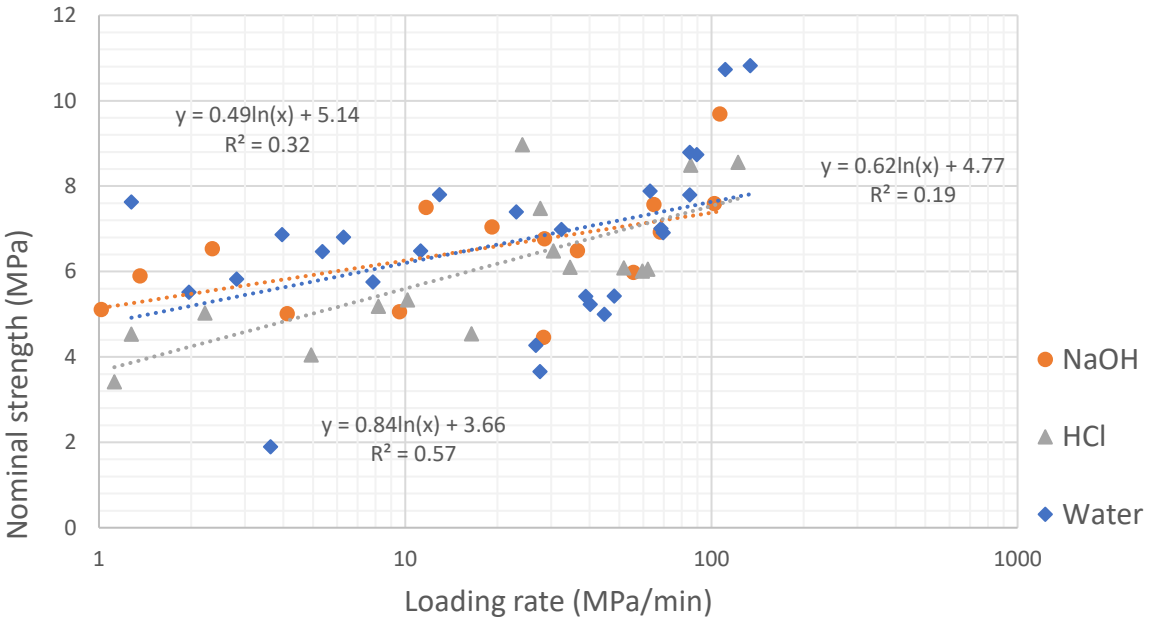


Figure 18 Water group compared with HCl and NaOH group

To examine the statistical significance of differences between groups, three *t* test evaluation was conducted to pairwise compare failure pressure of granites saturated by water, NaOH, and HCl. The significance level of HCl and NaOH group comparison is 0.374, which suggests that saturation by HCl and NaOH does not have a significant influence on the failure stress of granite. In addition, comparing the water group with the HCl group and NaOH group, respectively, gives the significance levels of 0.286 and 0.797, respectively, thereby showing that the HCl and NaOH are not significantly different from water. All three *t* test results indicate that the pH is not a statistically significant factor affecting the strength of granite.

At the same time, the possible influence of saturation time should also be considered. The two trend lines in Figure 19 show that the difference between short-time and a long-time saturation groups with NaOH is insignificant. Fluctuation of all data points covers essentially the same range,

which gives a similar slope and intercept. The comparison of saturation time with HCl, Figure 20, is slightly dissimilar from the NaOH case. It has a slight difference in slope and intercept, complementing the overall visual assessment that 24 hours of saturation leads to a very slight reduction of strength compared to 12 hours saturation. This suggests that the saturating time may have an influence on strength of granite.

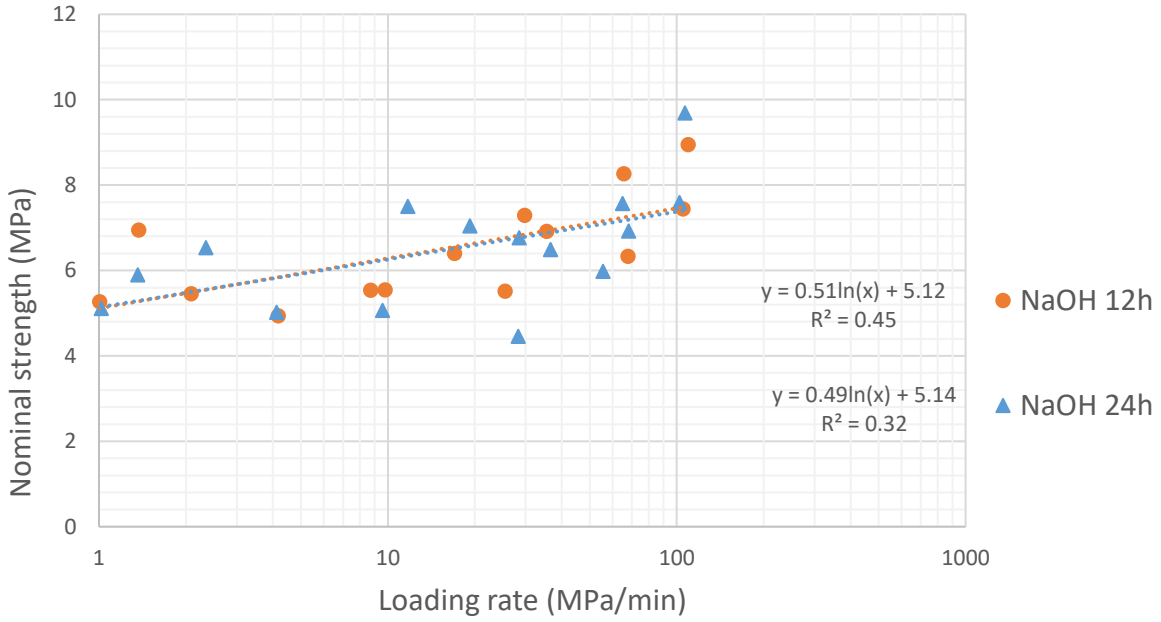


Figure 19 NaOH long-time saturation group compared with short-time saturation group

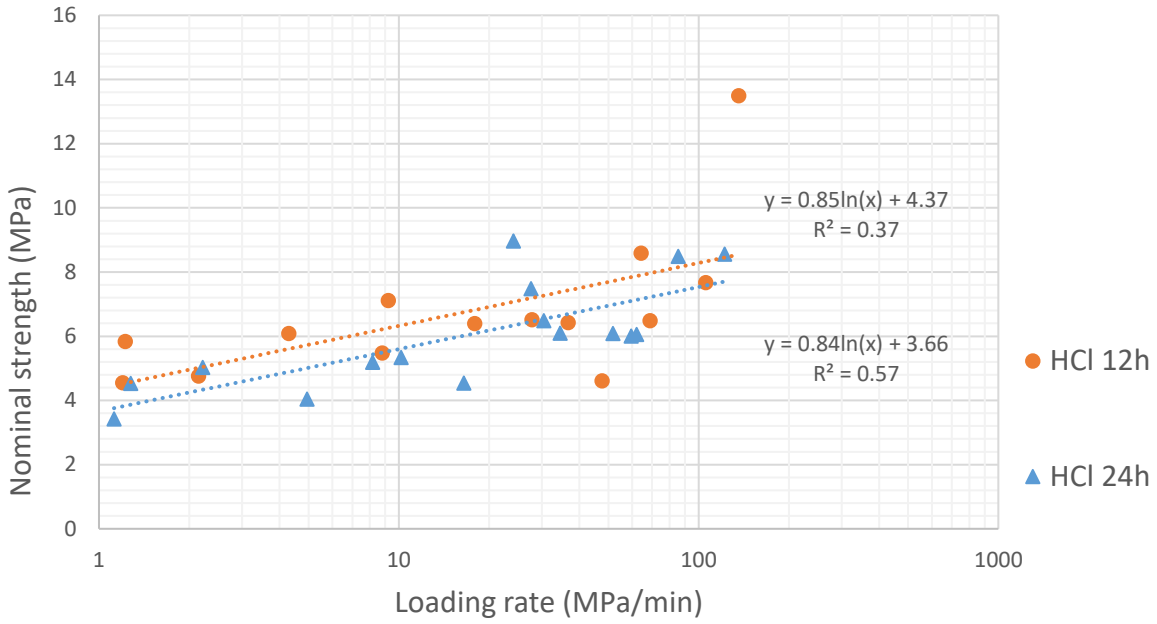


Figure 20 HCl long-time saturation group compared with short-time saturation group

To quantify the statistical significance of differences, *t* test evaluation is applied to analyze the influence of saturation. It gives the significance level of 0.938 and 0.341, respectively, for comparing long-time and short-time saturation groups of NaOH and HCl, respectively. The results show that time indeed has no statistically significant impact for either case, but the impact is a bit higher for HCL, albeit rather weak (less than 70% confidence that the two groups are from different populations).

4.4 KEROSENE VERSUS WATER COMPARISON

Figure 21 presents the data and trendline of kerosene and water. Here (as in other cases, strictly speaking) the trendlines are marginally meaningful because the data does not correlate to a line, but nonetheless, it is noted that the two groups have 53% difference in slope and 17% difference for intercept. More importantly, the data points still occupy the same range with a similar distribution. This suggests that the kerosene may have a similar ability to weaken the rock, as does the water. If indeed the two groups are the same, this result challenges the proposed mechanism of hydrolytic to weaken used in the past to explain the impact of water saturation on rock strength (Michalske 1982).

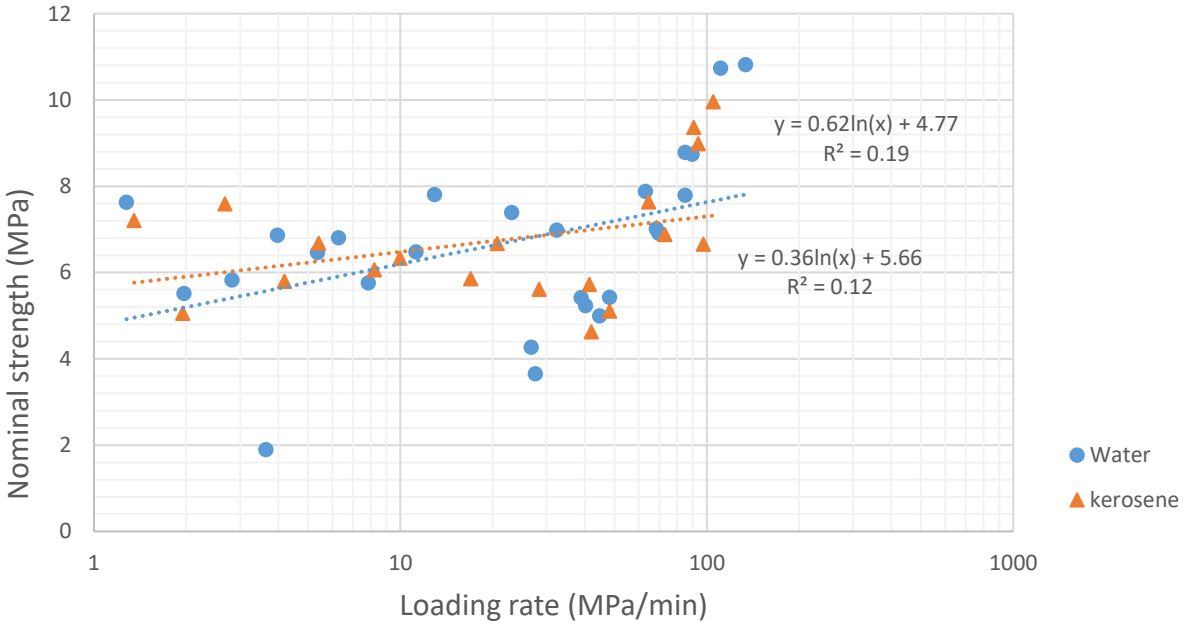


Figure 21 Kerosene group compared with water group

As with other cases, a *t* test was applied to compare the water and kerosene data results. The significance level of 0.9 shows that kerosene and water data is very close to one, which means they are statistically very similar. Thus, based on this result, it is reasonable to say that kerosene and water have a similar influence on strength of the granite tested in this study.

5.0 DISCUSSION

This chapter presents a discussion relating the experimental results to the original hypotheses. The first hypothesis is the strength of granite specimens saturated by fluid will be lower than the dry granite specimens, and it is tested by comparing the granite specimens saturated by several different fluids. Then hypothesized influence of the pH of the fluid is also tested by using water, 10% by weight sodium hydroxide, and 10% by weight hydrochloride acid. Finally, the last hypothesis, which relates to the possible difference between the effect of polar and nonpolar fluid, is tested by comparing the water and kerosene groups. In the end, a revised hypothesis based on grain lubrication is proposed, and discussed in light of recent experiments performed in the same granite using hydraulic oil, a non-polar fluid with substantially higher viscosity than kerosene.

5.1 FLUID LUBRICATION EFFECT

Experimental data supports the hypothesis that the strength of granite saturated by the fluid is lower than the strength of dry granite specimens. It is clear to see that all types of fluid reduced strength of granite specimens. Among the fluids, the average failure stress change, which is the mean of all failure stress of one experimental group, is shown in Table 2, confirming that all fluids result in 24%-32% reduction in strength.

Table 2 Average failure stress change among the fluids

Fluid	Failure stress of dry specimen (MPa)	Failure stress after saturation (MPa)	Reduction ratio
Water	8.9	6.7	25%
HCl	8.9	6.0	32%
NaOH	8.9	6.5	27%
Kerosene	8.9	6.7	24%

5.2 PH EFFECT

The results of the experiments, at best, provide weak support the hypothesis that pH has an influence on strength of granite. From Figure 22, the hydrochloric acid, on average, reduce strength slightly more compared to other types of fluid. Specifically, this greater strength reduction of granite saturated by HCl is evidenced at low loading rates. However, a significance level of the *t* test among the fluids shows that the pH does not have a significant effect on the strength of granite; there is an 80% probability that the NaOH and water groups are the same, and a nearly 30% probability that that HCl and water groups are the same. That being said, HCl consistently has the highest confidence of differing from other fluids (see Figure 22) and so, in this regard, HCl is different from other fluids and so the hypothesis that pH will have an impact is weakly supported.

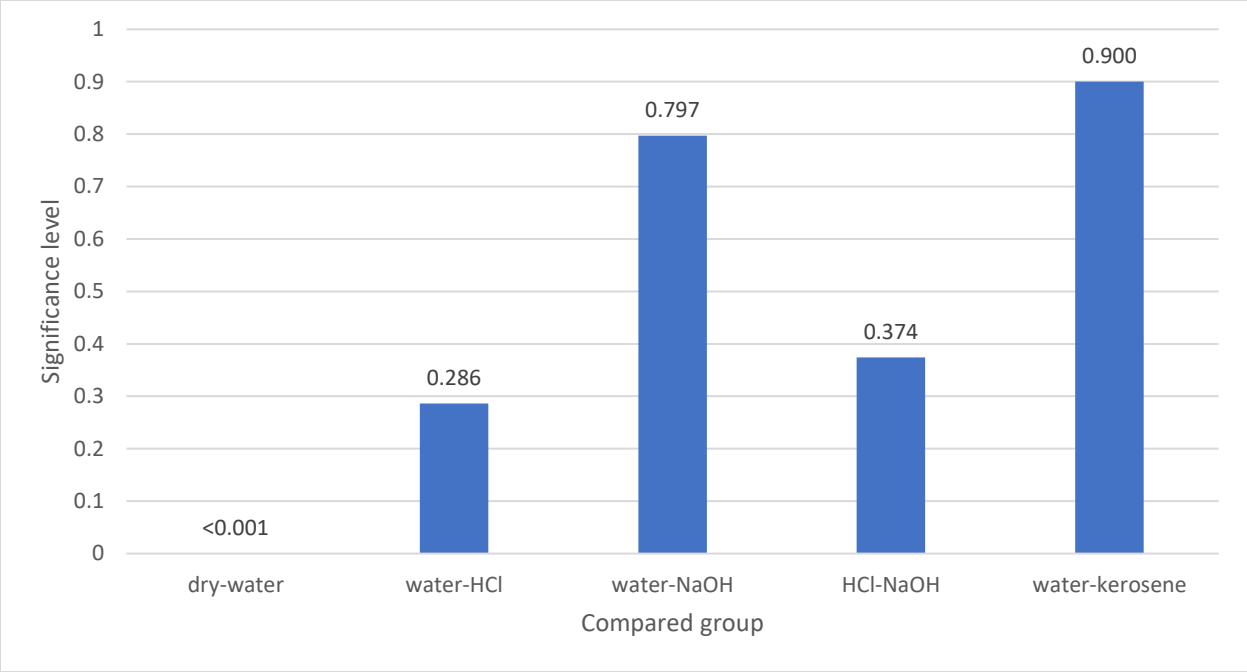


Figure 22 Significance level among fluids

For context, previous research found that strength of silica glass is increased when the pH of the fluid saturating increased (Mould 1961, Wiederhorn 1973). Mould (1961) observed that the strength increased when pH is greater 13 and decreased when pH is less than 1, and he further proposed that the pH influence is not primary, changing the strength in a range of ±10% with 6 mol/L HCl and NaOH. Langitan (1970) had a similar observation with Mould (1961) that validated the original finding. Considering the current experiment in light of these, the acid and base fluid used is 10% concentration by weight, which corresponding to 2.9 mol/L HCl and 2.8 mol/L NaOH that may lead to a smaller influence but with a similar trend. However, it observed from the new data that only the HCl reduces strength of granite, and only weakly and only in a range of low loading rate (1 – 20 MPa/min). Meanwhile, NaOH did not enhance as much strength as in the experiments in glass (Mould 1961, Wiederhorn 1973). The trend line in Figure 18, which is

marginally meaningful, shows a very small increase in strength at low loading rate relative to water, but the data points from water and NaOH groups still fall in the same area and *t* test shows that they are highly similar.

Since silica is a major chemical component of granite, it is reasonable to think that the chemical reaction should be similar with the reaction in silica glass. But, the results of the experiments in granite are only partly, and then only weakly, consistent with the previous research on silica glass. This implies important differences between silica glass where the Si-O bond is clearly the main mechanical unit associated with the strength, and granite, which is a chemically-heterogeneous crystalline rock that ostensibly held together not only by molecular bonds, but also by a kinematically-constrained (i.e. interlocking) grain structure (Potyondy 2004). Hence, the Si-O bond, and perhaps by extension any other molecular bond, is implied to be far less important in granite than in chemically and morphologically simpler materials.

5.3 POLAR AND NONPOLAR FLUID EFFECT

In the comparison of strength reduction of granite saturated water and kerosene, it shows that kerosene reduces strength very similarly to water. This result does not support that the polar fluid has more significant strength reduction than non-polar fluid on granite specimens. The average reduction of kerosene and water is 24% and 25% respectively, hence there is only a1% difference in the data. Additionally, the significance level in the *t* test is 0.9 showing that they have no significant difference. On the contrast, the experimental results are identical with 90% confidence. Charles (1958) and Wiederhorn (1967) found that the silica glass showed a strength reduction in an aqueous environment, and later Wiederhorn (1973) found the glass is also weakened in an

acidic solution environment. The results from previous experiments support that water and the water-based solutions have a strength reduction impact on a rock whose majority portion is silica. Later, interpretation of experiments on stress corrosion of glass in aqueous environments rely on an understanding that silica bonds are broken by water under stress (Ito 1981). In the water molecular, the hydrogen side holds a positive charge, and the other side of water molecule has a negative charge. When Silica is strained under application of stress, a hydrogen atom in free water connects to an oxygen atom in the Si-O bond, and then the Si atom in the strained silicon-oxygen bond attaches to instead an O-H in a free water molecule from the environment (Michalske 1982). The simple reaction is represented by (Ito 1981):

$$\mathrm{Si-O-Si} + \mathrm{H-OH} \rightarrow \mathrm{Si-OH} + \mathrm{Si-OH} \quad \textbf{(5-1)}$$

Because of the prevalence of Si-O bonds in silicate-rich rocks like granite, it is easy to assume that a similar mechanism should lead to weakening of granite by water that would exceed the weakening by a non-polar fluid with otherwise similar physical properties (such as density and viscosity). In this paradigm, kerosene is a mixture whose major compositions are hydrocarbons, so that kerosene is not a polar fluid and should not connect with strained silica. Therefore, when considering stress corrosion, granite saturated with kerosene should be stronger than granite saturated with water. But the experimental results do not support this hypothesis. The strength of granite reduction by water is essentially identical to the strength reduction by kerosene.

Similar to the discussion of the near independence of the strength reduction from the pH of the fluid, the observed complete lack of difference between water and kerosene calls into question the role of chemical bonding – and bond breaking – in the breakage of granite. Also, it can serve to

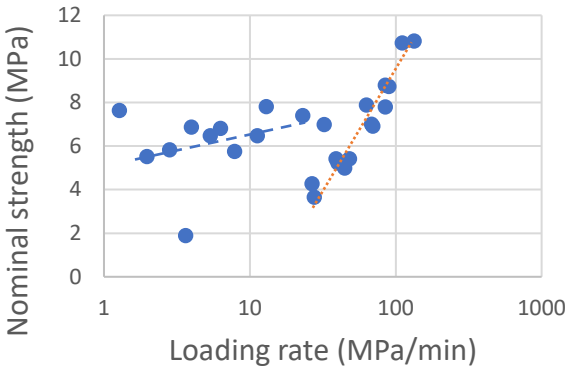
highlight consideration of the structural differences among granite, glass, and rocks used in past experimental studies. In the simple system silica glass, Mould (1961) stated that the strength of glass is reduced maximum 48.3% by 0.5% relative humidity, and maximum 63.6% by liquid water. Considering a sandstone that is 90% quartz, the strength is only decreased by 33% by addition of water (Feucht 1990). Granite also contains an approximate 75% of silica, which should highly influence the strength of granite if similar chemically-driven mechanisms are responsible for failure. Viewed on its own, the stress reduction of 25% by addition of water would seem to support this hypothesis. But, the same reduction is obtained by using kerosene. Hence reactions involving aqueous solutions and silica (including silica dissolution), appear to not significantly influence granite strength. Hence, the strength must be tied to something else. In the following section, a new hypothesis is proposed and initially tested wherein the strength of granite is primarily associated with the interlocked grain structure, and hence strength reduction is primarily achieved by lubricating between grains, making their relative movements less energetically dissipative and therefore requiring less energy to induce material failure.

5.4 VISCOSITY EFFECT

The impact of fluid on the intergranular mechanisms such as sliding has also been proposed to underlie some of the observed impact of fluid on the strength of rocks. The idea is that, fluid flows into pores of rock, forming a fluid film between the grains and decreasing the friction between grains (e.g. Menéndez 1996). Ogata (2004) observed that water reduced the strength of sandstone by 51% and proposed this is primarily due to intergranular friction reduction. A later experimental study on calcite fault gouge also showed the macroscopic frictional strength is reduced by water

(Violay 2014). Meanwhile, a recent study on limestone weakened by water was reported by Lisabeth (2015), which also shows evidence of impact of water on frictional strength. But, in these past studies, separation between chemical and lubrication effects is not clear because the saturating fluid was always water-based.

The experiments carried out in this study on granite provide initial indication that chemical effects are not particularly strong and, leaving it to be proposed that grain boundary lubrication is a primary mechanism whereby the fluids impact the strength of granite. Two observations are here presented that are relevant to this proposed hypothesis. Firstly, the data from granite specimens saturated by different fluids can be observed to be divided into two parts. As shown in Figure 23, the low rate and high rate portions of the data for all fluids can be fit by a trend, showing that the rate dependence of the strength is small for small loading rates and relatively stronger for higher loading rates (noting the loading rates are still well-below velocities approaching the wave speed of the material whereby inertia effects would become important). That is to say, in each graph, the dotted line in the second part is much steeper than the dashed line. However, this strong rate dependence did not observably appear in dry experiment group. In other words, this distinction of fast and slow loading behavior appears to somehow tie to the presence of fluid.


Nominal strength (MPa)
12
10
8
6
4
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10
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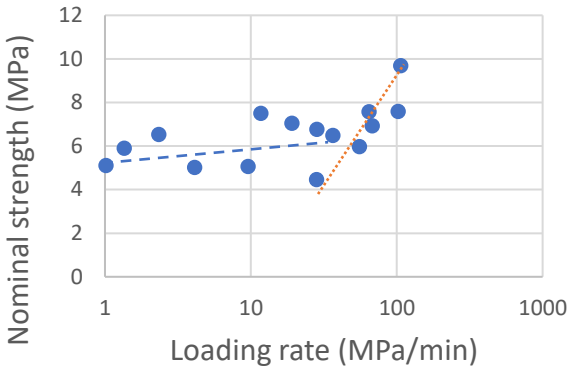
Loading rate (MPa/min)

(a)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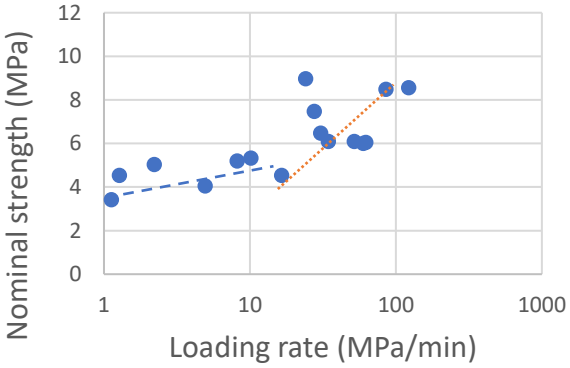
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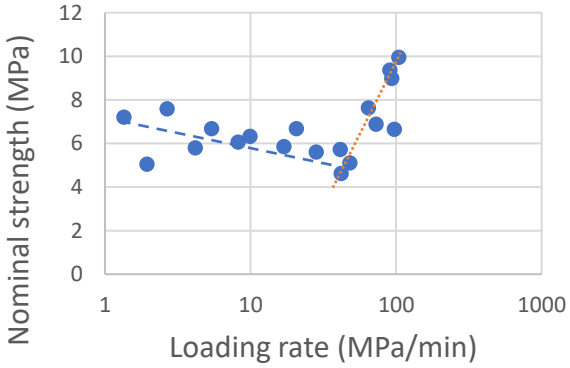
(c)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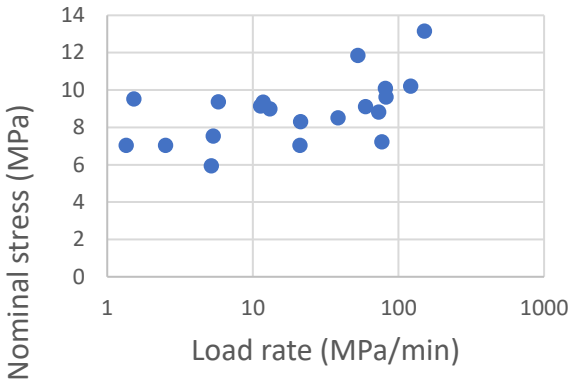

(e)

Figure 23 Comparison of granite saturated by various fluids.

(a) Water. (b) NaOH. (c) HCl. (d) Kerosene. (e) Dry

Because the strong rate dependence appears when applying a high loading rate, a possible reason could be a change of lubrication behavior with loading rate. That is to say, at low loading rates, there may be little impact of viscous flow between the grains because the shear rate is very small. But, at higher rates, the shear rate between the grains could be large enough that appreciable viscousity-related shear stresses are developed in the lubricating fluid layer.

Hence, with this understanding that the viscosity is one of the most important factors influencing the lubrication, it is reasonable to propose that the viscosity of the fluid may influence the position of the steep line. The viscosity of fluids used in current research range from 0.5 cp to 2 cp. This is a very small range and the values themselves are fairly low among fluids that are intended as lubricants. Ratoi (1999), for example, suggest that fluids with low viscosity will not be as effective in sustaining a lubricating fluid layer as larger viscosity fluids.

The proposed role of viscosity is initially tested by repeating the experiments using hydraulic oil (Jiangnan Zheng, University of Pittsburgh, Personal Communication 2018). The viscosity of the hydraulic oil used in the experiment is 54 cP, which is much higher than the other fluids used in this study. The resulting comparison among water, kerosene and hydraulic oil is shown in Figure 24. The first striking observation is that the strength of granite is lower with the hydraulic oil than with the other fluids. A *t* test is indicates the significance level is 0.030 and 0.005 for comparing hydraulic oil with water and kerosene, respectively. That is to say, the hydraulic oil can be considered to weaken the rock with more than 97% and 99% confidence, respectively. Such a result could suggest that increasing viscosity of the fluid gives a more effective lubrication film, which results in easier relative motion of the grains then eventually leads to failure at lower stress. While these observations provide some initial support to an hypothesis that the bulk lubrication is important to the strength of granite, it leaves some open questions as well. Firstly,the rate dependence of the strength is not as pronounced for hydraulic oil as for the lower viscosity fluids. So, a proposed origin for the rate dependence based on viscous stresses is perhaps not correct. Additionally, tying granite strength to grain boundary motions rather than molecular bond breakage raises problems when it comes to explaining time-delayed failure of granite under constant, subcritical stresses (e.g. Lu 2015). This so-called pheonomenon of static-fatigue has typically been cast in a kinetic fracture theory that arises from molecular bond breakage (after the seminal work of Zhurkov 1984 and more recent extention of Bažant 2009). Reconciling these new experimental results that seem to evidence little dependence on molecular bond breakage with experimental results demonstrating rate dependent fracture of granite consistent with an Arrhenious-type rate law presumably derived from bond breakage kinetics remains a challenge motivating ongoing research.

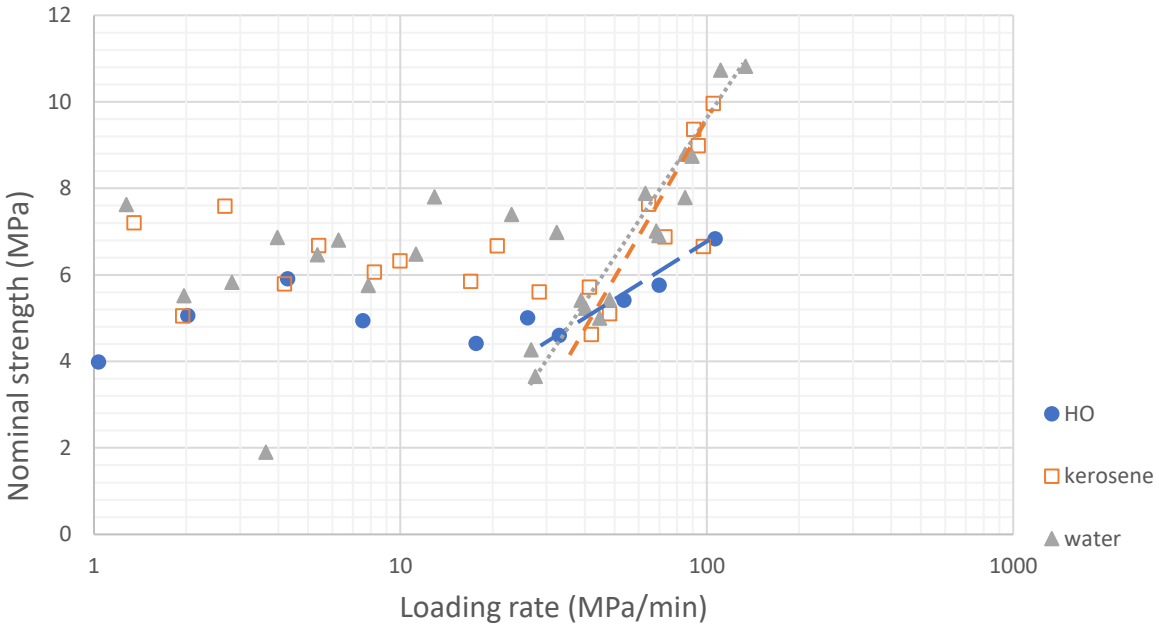

Figure 24 Hydraulic oil group compared with kerosene group and water group

6.0 CONCLUSIONS

The goal of this thesis is to investigate the impact of fluid on strength of granite, including the influence from water, base, acid, and nonpolar fluid. The experimental method is based on strength testing using a three-point flexural test with dry granite specimens and granite specimens saturated by four types of fluid. The fluids, including water, hydrochloric acid, sodium hydroxide, kerosene, divided the experiments into three comparison groups, and the results of these experiments can be summarized as follows:

1) At any pressure loading rate in the range from 1 to 150 MPa/min, the strength of granite specimens saturated by a fluid is lower than the dry granite specimens. On average, the strength of granite saturated by water, hydrochloric acid, sodium hydroxide, and kerosene decreases 25%, 32%, 27%, and 24%, respectively.
2) The pH of the saturated fluid has a minor influence on the strength of granite. The impact diminishes while the pressure loading rate is increasing.
3) For the granite specimens saturated by (nonpolar) kerosene, the strength is decreased similar to other fluids and essentially identically to water.
4) The significance level of HCl, NaOH, and kerosene compared with water is 0.286, 0.797, and 0.900 respectively, which indicates that the pH and polarity does not have statistically significant impact on strength of granite. But comparing the difference of significance level among fluids, it is observed that HCl is much closer

to the statistical significance than NaOH and kerosene, thereby suggesting the strength of granite may be more sensitive to the acidic fluid.

The striking overall conclusion of the research is that hypotheses developed based on an understanding of granite strength to arise primarily from chemical bonding, and specifically Si-O bonding, tend to fail, while hypotheses developed based on an understanding of granite strength to be connected to interparticle contacts tend to be supported by the data. As a first extension, a hypothesis that higher viscosity fluid will be more effective in lubricating grain boundaries and will therefore be led to greater strength reduction than lower viscosity fluids is supported by preliminary data. These results motivate ongoing research to quantify the trend by which increasing fluid viscosity can further weaken granite. Additionally, the role of fluid chemistry and viscosity under so-called static fatigue conditions, that is, where a constant load is held until failure, remains both untested and important for the purpose of resolving an apparent conflict wherein static fatigue behavior is classically tied to molecular-scale kinetics while this study finds the chemistry to apparently have little impact on the strength of granite.

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